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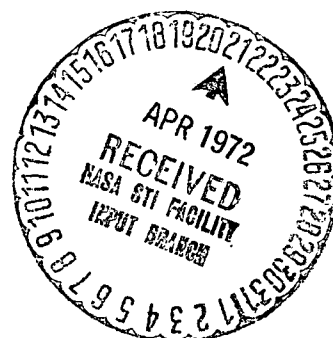
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METALLIC DISPERSIONS AS SOLID SAMPLES IN
ATOMIC ABSORPTION

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ABSTRACT. Colloidal dispersions of metals are obtained by using the metal samples as electrodes in an oscillating spark discharge; the compositions of the resulting highly stable and finely dispersed colloids correctly represent the compositions of the electrodes. These dispersions can be analyzed directly by atomic-absorption spectrometry with conventional apparatus. The error is estimated to be $\approx 1\%$. Two steels were analyzed for Cr by this method; the contents found were 16.19 and 15.99%, respectively, compared with 16.35 and 16.15% obtained by the standard chemical method. For the best precision, the samples are weighed before and after the spark discharge. The method can also be applied to nonmetallic conductors.

When demands for speed of operation, inversion economy, /123**
high level of sensitivity, and great versatility are important, the flame spectrophotometry method is without doubt the most appropriate. However, in metallurgical applications, the elapsed time between taking the sample and obtaining the analytical data is usually of primary importance. It limits the application of the method, since only solutions of the samples are used.

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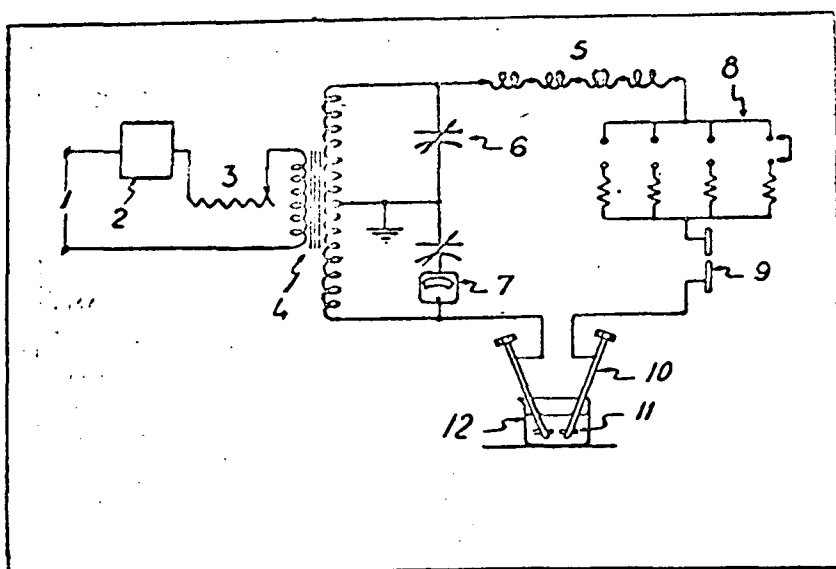
^{**} Numbers in the margin indicate the pagination in the original foreign text.

Obtaining these is usually a tedious operation, which should be done by specialized personnel, consuming incomparably greater amounts of time than those required for the subsequent analysis in the instrument.

To eliminate this difficulty, the steel industry uses costly, highly specialized spectrographs called direct-measurement instruments. In this article, a different solution is suggested for the problem of preparation time of the samples, which makes it possible to benefit from the advantages of flame spectrophotometry in obtaining the very fine metallic dispersions which may be directly analyzed in flames.

The preparation of colloidal suspensions of some noble metals using an arc under water has been known for many years (Bredig method, etc.), but the dispersions are useless for analytical purposes as formation of a large, thick deposit is unavoidable. Through the use of oscillating sparks, produced by properly stabilized discharges from condensers, very fine dispersions are achieved which are acceptable and highly stabilized. In the cases we studied, the composition of the dispersed material correctly represented the composition of the electrodes used, and they may be diluted as necessary. The dispersion operation is accomplished utilizing currents on the order of six amperes and at the rate of some ten milligrams per minute. They can be performed up to half an hour with refrigeration. Very concentrated dispersions are rarely used, unless it is desired to analyze components which appear as traces. Normally, dispersions obtained in times varying between four and eight minutes are employed. The electrodes we used are cylindrical, five millimeters in diameter, and ten millimeters in length.

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| 1 - Connection to the net. | 7 - Ampere meter (radiofrequency). |
| 2 - Autotransformer. | 8 - Resistances (0.3 - 3.0 ohms). |
| 3 - Limiting resistance. | 9 - Auxiliary electrodes. |
| 4 - 30 kV - 7.5 kVa transformer. | 10 - Electrode holders of the disperser. |
| 5 - Inductance, 0 - 300 microhenries. | 11 - Analytical electrodes. |
| 6 - Condensers, 0.01 - 0.001 microfarads. | 12 - Receptacle for the dispersions. |

ANALYSIS OF TWO TYPES OF STEEL BY ATOMIC ABSORPTION ON
SUSPENSIONS OBTAINED THROUGH ELECTRODISPERSION

"N.B.S." designation of standard steel	Chromium (%) according to "N.B.S." analysis	Chromium (%) determined in electrodispersions	Arithmetical means	Estimated error
S.S. 70	16.35	16.18 16.15 16.23	16.19	1.0%
S.S. 72	16.15	16.12 15.99 15.86	15.99	1.0%

DATA USED FOR INTERPOLATION

"N.B.S." designation	Chromium (%) according to "N.B.S."
S.S. 69	12.40
S.S. 71	24.00

When the dispersions are made for precision analysis, the electrodes are weighed before and after the attack by the spark. However, if the acceptable degree of precision is on the order of one or two percent, and, of course, using identical matrix standards, it is possible to control the attack by replacing the weighing

by an exact control of the time. The attached table corresponds to the analysis of stainless steel, performed with certified samples. It gives the determined chromium contents as well as those measured with five minute dispersions. The figure presents an outline of the source and the electrode-holding pincers.

In general terms, from an examination of the results, the following conclusions may be formulated: 1) the dispersion method using oscillating sparks may be applied generally to metals and their alloys. 2) The dispersions are colloidal in appearance and are highly reproducible. 3) The analysis of these dispersions, in the cases studied, demonstrated that it is representative of the composition of the electrodes. 4) The results are obtained regularly and rapidly, and it is occasionally possible to eliminate the weighings. 5) The excitation in the flame is produced with an efficiency comparable to that of true solutions. 6) The method may be extended to nonmetallic, but conductive samples. 7) The cost of the auxiliary equipment is not more than thirty or forty percent of the cost of a good flame spectrophotometric instrument.